

Republic of South Africa EDICT OF GOVERNMENT

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SANS 6170 (2010) (English): Water - Cobalt content



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Edition 1.2

SOUTH AFRICAN NATIONAL STANDARD

Water — Cobalt content



Edition 1.2

Table of changes

Change No.	Date	Scope
Amdt 1	2005	Amended to change the designation of SABS standards to SANS standards and to update referenced standards.
Amdt 2	2010	Amended to update referenced standards.

Foreword

This South African standard was approved by National Committee SABS SC 147A, *Water – Water sampling and analysis*, in accordance with procedures of the SABS Standards Division, in compliance with annex 3 of the WTO/TBT agreement.

This document was published in April 2010.

This document supersedes SANS 6170:2005 (edition 1.1).

A vertical line in the margin shows where the text has been technically modified by amendment No. 2.

Water — Cobalt content

1 Scope and field of application

This standard specifies a method of determining the cobalt content of water and wastewater, using an air-acetylene flame and direct flame atomic absorption.

The method is applicable to the determination of cobalt in the concentration range 0,1 mg/L to 10 mg/L.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies. Information on currently valid national and international standards can be obtained from the SABS Standards Division.

Amdt 1

SANS 111/ISO 835, Laboratory glassware – Graduated pipettes.

Amdt 1; amdt 2

SANS 112/ISO 648, Laboratory glassware – Single volume pipettes.

Amdt 1

SANS 115/ISO 385, Laboratory glassware – Burettes.

Amdt 1; amdt 2

SANS 128/ISO 1042, Laboratory glassware – One-mark volumetric flasks.

Amdt 1

SANS 3696/ISO 3696, Water for analytical laboratory use – Specification and test methods.

SANS 6168, Water – Pretreatment for metal analysis.

3 Principle

The pretreated sample is aspirated into an air-acetylene flame under oxidizing conditions and the absorbance measured at 240,7 nm is compared with the absorbances of cobalt standards measured under the same conditions.

4 Reagents

NOTE Unless otherwise specified, only use water that complies with the requirements of SANS 3696 and reagents of analytical reagent grade.

4.1 Nitric acid (HNO₃)

Concentrated (d at 25 °/25 °C = 1,42).

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4.2 Acidified water

Containing 1,5 mL of HNO₃ (see 4.1) per litre.

4.3 Cobalt stock solution (1 000 mg/L)

- **4.3.1** Obtain a commercially available stock solution of cobalt (1 000 mg/L) of guaranteed quality, or prepare the stock solution as described in 4.3.2.
- **4.3.2** Dissolve 4,037 g of cobaltous chloride hexahydrate ($CoCl_2 \cdot 6H_2O$), previously dried in a desiccator for 24 h, in 200 mL of water. Add 1,5 mL of HNO₃ (see 4.1) and dilute with water to 1 000 mL in a volumetric flask.

4.4 Cobalt working stock solution (100 mg/L)

Pipette 10,0 mL of the stock solution (see 4.3) into a 100 mL volumetric flask and dilute to the mark with the acidified water (see 4.2).

4.5 Cobalt standard solutions (0,1 mg/L to 10 mg/L)

Prepare at least three cobalt standard solutions that will bracket the expected concentration of the sample by dilution of the working stock solution (see 4.4), using the acidified water (see 4.2) as the diluent.

5 Apparatus

- **5.1** Atomic absorption spectrophotometer, for use at 240,7 nm.
- **5.2** Air-acetylene burner, suitable for attachment to the spectrophotometer (see 5.1).
- 5.3 Cobalt hollow-cathode lamp.
- **5.4** Oxidant, air, dried and filtered.
- **5.5** Fuel, acetylene, standard commercial grade. (Replace the cylinder when the cylinder pressure drops to 700 kPa.)

CAUTION: Never allow the operating pressure of acetylene to exceed 105 kPa.

- **5.6 Pressure-reducing regulators**, for the supply of fuel and oxidant to the instrument at the appropriate levels recommended by the manufacturer.
- **5.7 Glassware**. Where applicable, only use burettes, pipettes and volumetric flasks that comply I with the requirements for class A items as specified in SANS 111, SANS 112, SANS 115 and SANS 128, as relevant.

 Amdt 1; amdt 2

6 Sample pretreatment

Pretreat the sample, a blank (see 4.2) and at least one standard in accordance with SANS 6168 in order to analyse for the appropriate form of metal, i.e. soluble, insoluble or total metal.

NOTE For the purposes of legislation or specifications relating to this method, pretreat the sample for recovery of total cobalt, unless specifically indicated otherwise.

7 Procedure

7.1 Instrument operation

NOTE The differences between makes and models of atomic absorption spectrophotometers make it impracticable to formulate detailed operating instructions. Follow the manufacturer's operating instructions for the specific instrument but, in general, proceed in accordance with the instructions given in 7.1.1 to 7.1.10.

- **7.1.1** Turn on the instrument, install the cobalt hollow-cathode lamp (see 5.3) and apply the current recommended by the manufacturer.
- **7.1.2** Set the wavelength at 240,7 nm and set the slit width in accordance with the manufacturer's instructions.
- **7.1.3** Allow the lamp to stabilize for approximately 10 min and then align it to obtain optimum transmission.
- **7.1.4** Adjust the fine control of the wavelength setting of the monochromator in order to ensure maximum response.
- **7.1.5** Install the air-acetylene burner (see 5.2).
- **7.1.6** Check and, if necessary, adjust the gas flow rates to ensure that they are at the levels specified for the instrument.
- **7.1.7** Turn on the air supply and then the acetylene flow, and ignite the mixture.
- **7.1.8** Ensure that the flame is oxidizing (non-luminous with a hazy blue inner core), and then zero the instrument in the absorbance mode.
- **7.1.9** Aspirate a standard that will give an absorbance exceeding 0,2, and optimize the burner position until the maximum absorbance value is recorded. The instrument is now ready for calibration and analysis.
- **7.1.10** After analysis, extinguish the flame by turning off the acetylene flow first. When the flame is extinguished, turn off the air supply.

7.2 Instrument calibration

- **7.2.1** Aspirate the analyte blank (see 4.2) and zero the instrument.
- **7.2.2** Aspirate each of the standard solutions (see 4.5), using the acidified water (see 4.2) to flush the nebulizer between aspirations, and record their respective absorbances.
- **7.2.3** Construct a calibration curve, using the standard concentrations and their respective absorbances.

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7.3 Sample analysis

- **7.3.1** Aspirate the pretreated blank, standard(s) and sample (see clause 6), using the acidified water (see 4.2) to flush the nebulizer between aspirations.
- **7.3.2** Record the absorbances and determine the concentrations by referring to the calibration curve as constructed in 7.2.3, or record the concentrations directly if an instrument with concentration mode is used.
- **7.3.3** Correct the results for the sample and the control standard(s) by subtracting the result for the pretreated blank, if applicable, and verify the absence of interference due to the pretreatment method by confirmation of the true concentration(s) of the control standard(s).

NOTE If the nitric acid digestion technique is used, absorbance enhancement may be experienced as a result of high nitrate levels. If positive interference is detected in the control standard, repeat the determination, pretreating all the standards with the sample and blank as directed in clause 6.

8 Expression of results

So express the result as to indicate the form of metal analysed and the concentration units used, e.g. 'Total cobalt as Co in mg/L'.

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SABS - Standards Division

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South African National Standards are updated by amendment or revision. Users of South African National Standards should ensure that they possess the latest amendments or editions.

The SABS continuously strives to improve the quality of its products and services and would therefore be grateful if anyone finding an inaccuracy or ambiguity while using this standard would inform the secretary of the technical committee responsible, the identity of which can be found in the foreword.

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